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Applicants: Stoyanov et. al.

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Title: Method For Forming Individualized Intrafiber Crosslinked Cellulosic Fibers With Improved Brightness and Color

DECLARATION OF ANGEL STOYANOV PURSUANT TO § 37 C.F.R. § 1.132

Federal Way, WA,  
August 16, 2006

TO THE COMMISSIONER OF PATENTS:

I, Angel Stoyanov, declare and state as follows:

1. I am currently employed by the Weyerhaeuser Company as a Scientist and since 1998 have worked exclusively on crosslinking of cellulosic fibers.

2. I received my Bachelor of Science and my Master Of Science from the University of Chemical Technology and Metallurgy at Sofia, Bulgaria, in 1980 and 1981, respectively. After graduation my work history is as follows:

I was a Research Assistant from 1982 to 1986 and an Assistant Professor from 1986 to 1994 at the University of Chemical Technology and Metallurgy at Sofia, Bulgaria. From 1990 to 1991 I worked under a Fulbright scholarship at the University of Washington, Seattle, WA, and completed all graduate courses for a Ph. D. in 1996. From

1996 to 1998 I conducted research for my Ph. D. and held various teaching positions in the Department of Engineering at the University of Washington.

3. I have read and am familiar with the Hansen et al patents US Patent No. 5,589,256 and 5,789,326

4. Hansen et al state in the '256 patent that initial application of the binder on high bulk fibers preferably occurs after the curing step, particularly if the binder is capable of functioning as a crosslinking material. Hansen then states that specific binders that can also crosslink are polyols, polycarboxylic acids and polyamines. If such binders are present during curing, the binder will be consumed during the curing step to form covalently crosslinked bonds. When this occurs, the binder is no longer available for hydrogen bonding or coordinate covalent bonding, and particle binding to fibers is ineffective, column 23, line 4-14.

5. Tests were undertaken to determine if polyols indeed act as crosslinking agents with cellulose. Accordingly, I planned and supervised experiments which were carried out by my technician Derik Rieger.

6. Exhibit A shows the experimental design for the tests. All samples were cured at 171°C for 7 minutes. The acronyms are as follows: COP, chemical on pulp (CF416 pulp from Weyerhaeuser Co.); SHP, sodium hypophosphite; CA, citric acid; SOR, sorbitol; and XYL, xylitol. Exhibit B shows the addition levels for the various reagents; Exhibit C gives the procedure, Exhibit D shows the results of brightness testing by TAPPI T 525 om-02 and Exhibit E, the FAQ wet bulk results determined by the procedure in the application. The Hunter color values were determined by TAPPI T 1231 sp 98. Whiteness Index, WI<sub>(CDM-L)</sub>, was calculated from the formula, WI<sub>(CDM-L)</sub> = (L-3b).

7. The results are summarized in Table 1.

Table 1

Fiber Properties

Sample	Wt. % on Dry Fiber						ISO Brightness			Hunter Color		WI <sub>1(CDM-L)</sub>
	CA	SHP	Sorbitol	Xylitol	FAQ Bulk, cc/g	Wet %	L	a	b	L	a	
A	0	0	0	0	11.59	82.7	94.9	-0.83	5.58	78.16		
B	0	2	0	0	12.26	82.8	95.0	-0.83	5.58	77.87		
C	8	2	0	0	18.48	78.5	94.7	-2.02	8.67	68.69		
D	8	2	2	0	18.29	83.7	95.3	-1.41	5.53	78.71		
E	8	2	6	0	17.05	85.4	95.7	-1.23	4.80	81.3		
F	8	2	0	2	18.18	84	95.6	-1.45	5.7	78.50		
G	8	2	0	6	16.83	85.8	95.7	-1.21	4.53	82.10		
H	0	2	2	0	11.43	82.3	94.8	-0.88	5.81	77.37		
I	0	2	6	0	11.10	81.4	94.4	-0.81	5.96	76.52		
J	0	2	0	2	11.27	80.5	94.1	-0.78	6.20	75.50		
K	0	2	0	6	10.76	79.8	93.3	-0.76	5.60	76.50		

8. It is well recognized by those skilled in the art of crosslinked fibers that an increase in FAQ wet bulk, relative to an untreated control, reflects that fibers have been crosslinked.

9. Sample A is a control and Sample B is the pulp with 2 percent by dry weight sodium hypophosphate; FAQ wet bulk values are 11.59 and 12.26 cc/g, respectively, and  $WI_{(CDM-L)}$  values are 78.16 and 77.87, respectively. When pulp is treated with citric acid and sodium hypophosphate, Sample C, FAQ wet bulk is 18.48 cc/g and the Whiteness Index is 68.69. When pulp is treated with citric acid, sodium hypophosphate and sorbitol, a polyol, at the 2 and 6 percent by weight level of sorbitol on pulp, Samples D and E, respectively, FAQ wet bulk is significantly increased to 18.29 and 17.05 cc/g, respectively. The Whiteness Index of Samples D and E, also increased to 78.71 and 81.30, respectively. However, when pulp is treated only with sodium hypophosphate and two different levels of sorbitol, 2 and 6 percent by weight, Samples H and I, there is no increase in FAQ wet bulk; Whiteness Index, decreased relative to the control pulp and the pulp sample with only sodium hypophosphate, Samples A and B, respectively.

When pulp is treated with citric acid, sodium hypophosphate and xylitol, a polyol, at the 2 and 6 percent by weight level of xylitol on pulp, Samples F and G, respectively, FAQ wet bulk is significantly increased to 18.18 and 16.83 cc/g, respectively. The Whiteness Index of Samples F and G, also increased to 78.50 and 82.10, respectively. However, when pulp is treated only with sodium hypophosphate and two different levels of xylitol, 2 and 6 percent by weight, Samples J and K, there is no increase in FAQ wet bulk; Whiteness Index  $WI_{(CDM-L)}$ , decreased relative to the control pulp and the pulp with only sodium hypophosphate, Samples A and B, respectively.

10. Based on the fact that there is no increase in FAQ wet bulk when pulp is treated only with sodium hypophosphate and sorbitol, or only with sodium hypophosphate and xylitol, it is my opinion that the polyol, sorbitol, and the polyol, xylitol, do not crosslink with cellulose.

11. In accordance with accepted Patent Office Practice, the dates in the laboratory notebook pages presented in Exhibits A- E have been redacted.

12. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

Respectfully submitted,

Date 8/16/06



Angel Stoyanov